
वायु प्रदूषण मापने की पद्धति
भाग 24 सूक्ष्म कणिय पदार्थ (पी एम्_{2.5})

Methods for Measurement
of Air Pollution

Part 24 Fine Particulate Matter (PM_{2.5})

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भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

FOREWORD

This Indian Standard (Part 24) was adopted by the Bureau of Indian Standards, after the draft finalized by the Air Quality Sectional Committee had been approved by the Chemical Division Council.

Particulate matter (PM) or particulates are microscopic solid or liquid matter suspended in atmosphere. They can be either anthropogenic or naturally occurring. Some examples include dust, ash and sea-spray. Particulate matter (including soot) is emitted during the combustion of solid and liquid fuels, such as from power generation, domestic heating and in vehicle engines. Particulate matter varies in size (that is, the diameter or width of the particle). PM_{2.5} means suspended particles that are 2.5 micrometers (µm) or smaller in diameter. It is often called as fine particulate matter.

Inhalation of particulate matter can have adverse health effect. The biggest impact of particulate air pollution on public health is understood to be from long-term exposure to PM_{2.5}, which increases the age-specific mortality risk, particularly from cardiovascular causes. Children, the elderly and those with pre-disposed respiratory and cardiovascular disease, are known to be more susceptible to the health impacts from air pollution. Keeping this in view, it is important to develop standards for determination of fine particulate matter present in atmosphere.

In the development of this standard, considerable assistance has been derived from the following:

Appendix L to Part 50 of USEPA Federal Register — Reference Method for the determination of Fine Particulate Matter as PM_{2.5} in the atmosphere.

ASTM F 316-94 Standard Test Methods for Pore Size Characteristics of Membrane Filters by Bubble Point and Mean Flow Pore Test

ASTM D2896a-99 Standard Practice for Evaluation of Air Assay Media by the Monodisperse DOP (Diocetyl Pthalate) Smoke Test

The list of committee members responsible for development of this standard is given in Annex A.

In reporting the results of a test or analysis in accordance with this standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART 24 FINE PARTICULATE MATTER (PM_{2.5})

1 SCOPE

This standard (Part 24) prescribes the method for measurement of the mass concentration of particulate matter (PM_{2.5}) in ambient air based on the principle of segregation of non-PM_{2.5} particles from the air stream followed by filtration and gravimetric determination of PM_{2.5} mass on the filter substrate.

2 REFERENCE

The following standards contain provisions which through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
4167 : 1980	Glossary of terms relating to air pollution (<i>first revision</i>)
5182 (Part 23) : 2006	Methods for measurement of air pollution: Part 23 Respirable suspended particulate matter (PM ₁₀) cyclonic flow technique
17025 : 2017	General requirements for the competence of testing and calibration laboratories (<i>second revision</i>)

It is further recommended to refer the following documents for more information:

- a) Quality Assurance Guidance Document 2.12: Monitoring PM_{2.5} in Ambient Air using Designated Reference or Class I Equivalent Methods, EPA.
- b) EPA Compendium Method IO 3.3.

3 TERMINOLOGY

For the purpose of this standard the definitions given in IS 4167 and the following shall apply.

3.1 Particulate Matter PM_{2.5} — Figure 1 in IS 5182 (Part 23), defines various size distributions of particulate matter in ambient air. Particulate matter having an aerodynamic diameter less than or equal to a nominal 2.5 micrometers is referred to as PM_{2.5}.

NOTE — It may however, be noted that the internationally accepted size distribution for PM_{2.5} particulates includes a small fraction of particles up to an aerodynamic diameter of less than 5 micrometers.

4 PRINCIPLE

An electrically powered air sampler draws ambient air at a constant volumetric flow rate of 16.67 lpm (1 m³/h), maintained by a mass flow controller coupled to a microprocessor, into a specially shaped inlet and through an inertial particle size separator (impactor) where, suspended particulate matter in the PM_{2.5} size range is separated for collection on a polytetrafluoroethylene (PTFE) filter over the specified sampling period.

Each filter is weighed (after moisture and temperature conditioning) before and after sample collection to determine the net mass of PM_{2.5} particles.

The total volume of air sampled is determined by the sampler at actual ambient temperature and pressure prevailing at the time of sampling.

The mass concentration of PM_{2.5} in ambient air is computed as the total mass of collected particles in the PM_{2.5} size range divided by the actual volume of air sampled and is expressed in micrograms per cubic meter of air (µg/m³).

The measurement process is considered to be non-destructive, and the PM_{2.5} sample obtained can be subjected to subsequent physical or chemical analysis.

5 RANGE AND SENSITIVITY

5.1 Minimum Detection Limit

The lower detection limit of the mass concentration measurement range is estimated to be approximately 2 µg/m³ based on noted mass changes in field blanks. It may be mentioned that the above limit assumes use of a standard micro balance with a resolution of 1 µg.

5.2 Upper Detection Limit

The upper limit of the mass concentration range is determined by the filter mass loading beyond which the sampler can no longer maintain the operating flow rate within specified limits due to increased pressure drop across the loaded filter. The upper limit is also a function of size distribution of particles, humidity and sampler flow rate control system. Nevertheless,

all samplers are expected to be capable of measuring 24 h samples with a $PM_{2.5}$ mass concentration of upto $200 \mu\text{g}/\text{m}^3$ while maintaining the operating flow rate within specified limits. For $PM_{2.5}$ concentration above $200 \mu\text{g}/\text{m}^3$ or environmental conditions of high humidity (above 80 percent RH), the sampler can be operated in two or three shifts of equal time duration by changing filter paper.

6 INTERFERENCES AND SOURCES OF ERROR

6.1 Filter Conditioning

Filter conditioning environments can lead to different mass measurements as a function of relative humidity (RH). Several species of $PM_{2.5}$ particles are known to be hygroscopic and can absorb substantial quantities of water especially above the deliquescence point. Humid conditions will therefore cause a positive bias. Some $PM_{2.5}$ fractions are known to be volatile and can be lost with few degrees rise in temperature. As such higher temperatures during operation or storage of the filters can lead to a negative bias. The temperatures during operation stage and during storage of the filters are to be maintained as per requirements given in 8.4 to avoid any bias.

6.2 Change of Oil Soaked Filter Paper

The $PM_{2.5}$ sampler has a well impactor ninety-six (WINS) designed to trap medium size particulates with an aerodynamic diameter between 2.5 and 10 microns on a 37 mm diameter GF/A paper immersed in silicone oil to avoid sampling errors due to the tendency of small particles to bounce off the impaction surface. The oil soaked filter paper shall be removed after every run.

6.3 Static Charge

Sensitivity of micro-balances can lead to artifact errors due to static charge buildup on the PTFE filters. Static charge can also be transferred to the filter from the clothes / body of the operator during handling. Good handling practices, for example, non-metallic, non-serrated forceps for handling weights, use of grounded wrist cuffs while handling the filters, antistatic, nitrate-free, phosphate-free, sulphate-free, and powder free vinyl gloves or use of appropriate devices for static charge removal, such as antistatic strips for static charge neutralization are recommended if filter weights vary by more than $\pm 0.2 \text{ mg}$.

6.4 Contamination with Ambient Dust

$PM_{2.5}$ samples often have a net mass of only few micrograms. As such filters from $PM_{2.5}$ samplers can be easily contaminated by even a few particles of settleable dust leading to a positive bias. To avoid such errors filters should be kept covered at all times, handled only with tweezers and loaded into filter carriers/cassettes in a clean lab environment as mentioned in 9.

6.5 Contamination during Filter Handling

$PM_{2.5}$ filters should be handled only with non-metallic, non-serrated blunt forceps to hold the filter only on the support ring and by wearing antistatic, nitrate-free, phosphate-free, sulphate-free, and powder free vinyl gloves. Handling filters with bare hands can leave residues on it leading to a positive bias. Contact with exposed surface of the filter can dislodge some of the dust deposited leading to a negative bias.

6.6 Contamination from Pump Exhaust Releases

Oil lubricated or rotary carbon vane suction pumps constantly release fine oil mist droplets and fine carbon particles (as carbon vanes are continually abraded) through their exhaust. These mists / droplets / fine carbon dust can get entrained into the sampler inlet and can cause a positive bias. Therefore, as mentioned in 7.2, sampler shall use only oil and carbon vane free pumps.

7 APPARATUS

7.1 Filter for $PM_{2.5}$ Sample Collection

Any filter manufacturer offering filters for collection of $PM_{2.5}$ samples shall conform to the following performance specifications, certified from an IS/ISO 17025 accredited laboratory.

7.1.1 Size

Circular (outer diameter), $46.2 \pm 0.25 \text{ mm}$. Direct outside diameter measurement with caliper.

7.1.2 Medium

Polytetrafluoroethylene (PTFE) with integral support ring.

7.1.3 Support Ring

Polymethylpentene (PMP) or equivalent inert material like perfluoroalkoxy (PFA), $0.38 \pm 0.04 \text{ mm}$ thick (measurement with micrometer). Width of $3.68 \text{ mm} + 0.00 \text{ mm}$, $- 0.51 \text{ mm}$ (measurement with caliper) all along the outer periphery of the filter membrane.

7.1.4 Pore Size

$2 \mu\text{m}$, measured as per method described in ASTM F316-94

7.1.5 Filter Membrane Thickness

30 to $50 \mu\text{m}$ (measurement with micrometer).

7.1.6 Maximum Pressure Drop

For a clean filter, measured maximum pressure drop shall be limited to 30 cm Water Column at 16.67 lpm air flow while measuring as per method prescribed in ASTM D2986a-99.

7.1.7 Collection Efficiency

Greater than 99.7 percent for 0.3 μm particles as measured by the DOP test at the samplers operating face velocity (see 7.1.6).

7.1.8 Filter Numbering:

It is advisable that each filter has a unique ID number printed on the support ring to facilitate keeping of initial & final weight records and other relevant sample information.

7.1.9 Maximum Moisture Pick Up

The filter shall not increase in weight by more than 10 μg after 24 h exposure to air of 40 percent RH at $27 \pm 2^\circ\text{C}$ compared to its weight after 24 h exposure to air of 35 percent RH at $27 \pm 2^\circ\text{C}$ (gravimetric).

7.1.10 Filter Weight Stability

The average filter weight loss shall be less than 20 μg as measured in each of the following two tests. Filter weight loss shall be the average of difference between the initial and the final filter weights of a random sample of test filters selected from each lot.

- a) *Test for Loose Surface Particle Contamination* — After the initial weighing, each filter shall be placed in a filter cassette and dropped from a height of 25 cm onto a clean particle free surface. Repeat two more times for a total of 3 drops. Each filter is then removed and reweighed. The average change in mass shall be less than 20 μg .
- b) After drop test is completed each filter is then exposed to a drying oven, at $40 \pm 2^\circ\text{C}$, for not less than 48 h. Remove, condition to room temperature and reweigh each filter. The average change in mass shall be less than 20 μg .

7.1.11 Alkalinity

Less than 25 micro-equivalents/gram of filter when tested as per the procedure prescribed in Quality Assurance Guidance Document 2.12 Monitoring $\text{PM}_{2.5}$ in Ambient Air using Designated Reference or class I equivalent methods.

7.1.12 Packaging

The filters shall be packed clear, hard, dust tight, non-contaminating plastic containers.

7.1.13 If filters are also to be used for metal analysis, these will be tested for metal contamination by X-Ray fluorescence as per EPA compendium method IO 3.3 and maximum concentration shall be, Pb - 4.8; Hg - 4.4; Cd - 10.8; As - 2.8; Cu - 2.8; Fe - 5.8; Mn - 2.2; Ni - 3.0 and Cr - 2.2 ng/cm^2 .

7.2 Sampler

The $\text{PM}_{2.5}$ sampler shall consist of a sample air inlet, downtube, particle size separator (impactor), filter

holder assembly, oil free carbon vane pump, air flow rate control system, flow rate measuring device, ambient and filter temperature monitoring system, barometric pressure measuring system, all housed in an outdoor environment enclosure. Fig. 1 shows a typical system configuration.

7.2.1 Recording Facility

The sampler shall have facility to record the air flow rate, total volume of air sampled, coefficient of variation of flow rate, ambient and filter temperature, barometric pressure and filter pressure prevailing during the operation of the sampler. The data of flow control must be upgraded at least every 30 s and recorded (logged) every five minutes. Barometric pressure, ambient temperature and filter temperature should be measured on the same schedule. Filter temperature must not exceed the ambient temperature by more than 5°C for more than 30 min. Blowing filtered ambient air through the enclosure provides the necessary cooling effect. The supporting run-time (interval data) generated after 5 min intervals as well as the integrated performance (filter) data for the 24 h sampling period shall be stored in the sampler's microprocessor with the facility to retrieve the same as and when required. The data may also be extracted to a portable computer.

7.3 Sampler Height

The sampler shall be equipped with means to maintain the sampler in a stable, upright position and such that the center of the air sample inlet is maintained in a horizontal plane at 2.0 ± 0.2 m above the floor. Suitable means like bolt holes, brackets /tie downs may be provided to mechanically secure the sampler from toppling over due to wind.

7.4 Dust Segregation System

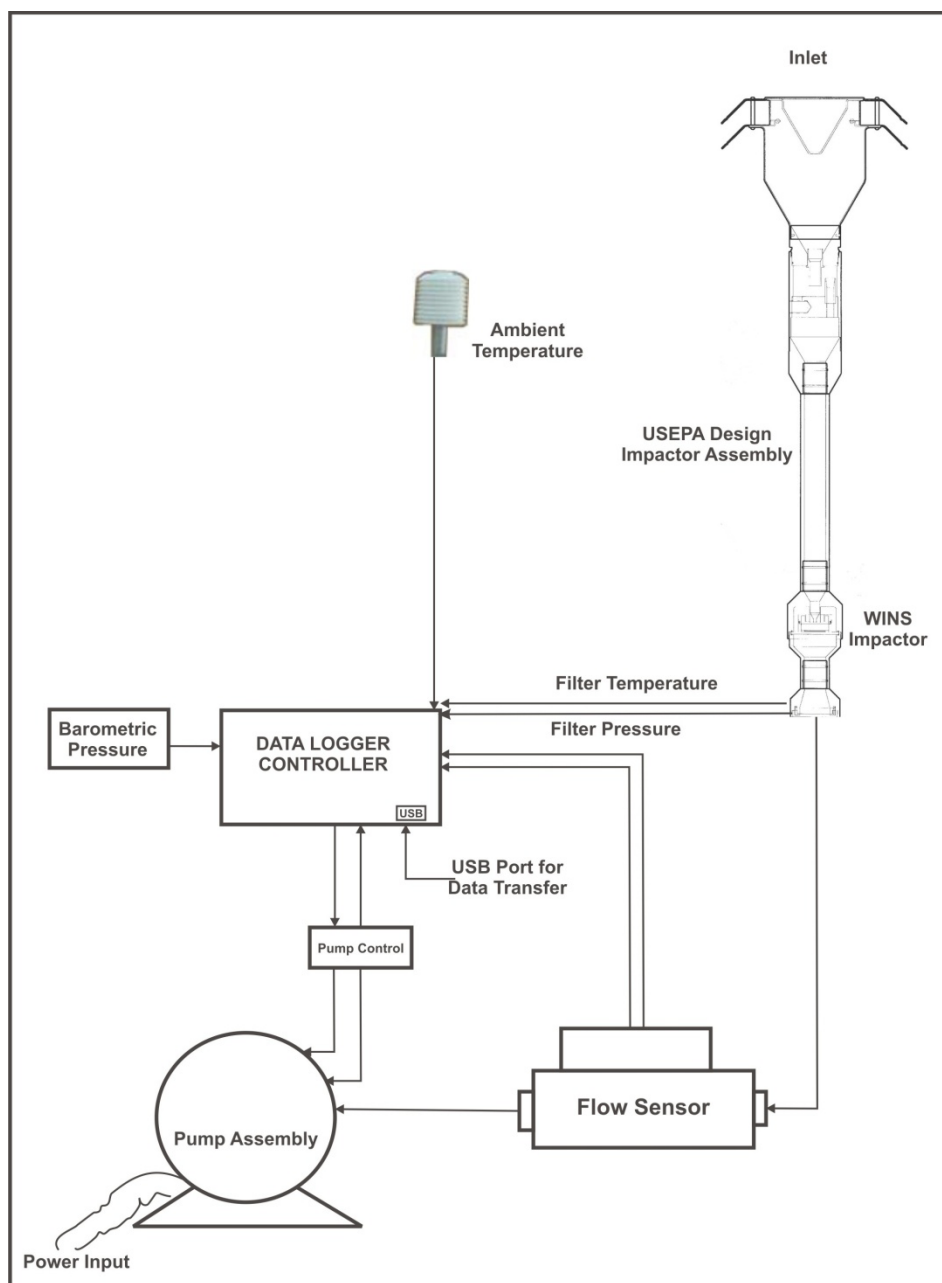
The dust segregation system shall comprise of two stage impaction separators connected by a downtube as indicated in Fig.1. The parts of the PM_{10} impactor and downtube shall be fabricated as per internationally accepted designs (refer Figures L-1 through L-19 of appendix L of 40CFR 50 of the USEPA federal register).

7.5 $\text{PM}_{2.5}$ Impactor

The $\text{PM}_{2.5}$ impactor (particle size separator) is the heart of the system and shall be fabricated to close tolerances as per internationally accepted designs to ensure effective size cut-off (refer Figures L-20 through L-24 of appendix L of 40CFR 50 of the USEPA federal register).

7.5.1 Impactor Filter

Impactor filter shall be circular in size, 35 to 37 mm diameter, made of glass fiber with pore size of about 1.5 μm , without binder.

FIG. 1 PM_{2.5} SAMPLER SCHEMATIC

7.5.2 Impactor Oil

The filter placed inside the oil well of the impactor shall be soaked with 1 ml single compound silicone oil having a vapor pressure of 2×10^{-8} mm Hg and viscosity 36 – 40 centistokes at 25°C.

7.5.3 Filter Holder Assembly

The sampler shall have a filter holder assembly connected to the lower portion of the PM_{2.5} impactor assembly, which can hold and seal the filters placed in filter cassettes. The filter holder shall be placed in

a horizontal position such that the sample air passes down through the filter at a uniform face velocity.

7.5.4 Leak Check Adaptor

The sampler shall include an accessory device that can be mounted on the downtube in place of the PM₁₀ impactor with a provision to completely seal/shut-off the air inlet into the system so that a leak check operation can be performed in the field without any additional hardware.

7.6 Analytical Balance

For precise and reliable measurement of mass of $PM_{2.5}$ in ambient air, use of digital microbalance with higher resolution is recommended (*see note below*). The installation of digital micro balance shall be as per manufacturer's instruction on a vibration damping surface.

NOTE — A digital microbalance with a resolution of 10 μg shall suffice for $PM_{2.5}$ levels encountered at large number of site conditions in India. However, if a laboratory wants to increase its range of measurement of $PM_{2.5}$ levels to cover the sensitive areas or other very low dust environments a digital balance with a resolution of 1 μg is recommended.

8 PERFORMANCE CRITERIA

8.1 Sample Flow Rate

For effective separation of particulate matter (that is, PM_{10} and $PM_{2.5}$) and performance of the impactor system, specific air velocities should be maintained through the device. The sample air flow rate of 16.67 l/min (1.00 m³/h), measured as actual volumetric flow rate using a mass flow controller at the temperature and pressure of the sample air entering the inlet, should be maintained at all times with an accuracy of ± 2 percent, through proper operation of the impactor.

8.2 Coefficient of Variation

The flow rate control system should be capable of maintaining flow rate with a coefficient of variation not exceeding 2 percent throughout the duration of operation of the sampler.

8.3 Flow Rate Cut-off

The sampler's air flow rate control system shall have provision to terminate operation of air sampler for the remainder sampling period in the event that the air flow rate deviates by more than 10 percent from the designed flow rate for a continuous period exceeding 60 s. However, this sample cut-off provision shall not apply during periods the sampler is inoperative due to temporary power interruption.

8.4 Filter Temperature Control

The sampler shall have suitable means to limit the temperature rise of the sample filter from insolation and internal dissipation sources by not more than 5°C above the temperature of ambient air surrounding the sampler during the entire sampling period.

9 PROCEDURE

9.1 $PM_{2.5}$ filter shall be inspected, conditioned and weighed in a clean lab environment that shall include separation to avoid cross contamination including controlled access for restricted movement of personnel. The samplers are not recommended to be stored

or maintained in filter inspection, conditioning or weighing area.

NOTE — Use of semi-wet methods to clean floors, work surfaces as per the regime established by laboratory and measures as removal of shoes before entry into the $PM_{2.5}$ filter handling area; use of dedicated lab coats inside; cleaning of hands at entry; use of floor mats or air curtains or double door entries as decided by lab depending upon site conditions.

9.2 Suitable arrangement like light box shall be available for inspection of filter. Filters with following defects shall be discarded:

- Pinhole* — Any small hole appearing as a distinct and obvious bright point of light when examined over a lighted table.
- Separation of Ring* — Any separation or lack of seal between the filter and the filter support ring.
- Chaff or Flashing* — Any extra material on the reinforcing ring or on the heat-seal area that would prevent an airtight seal during sampling.
- Loose Materials* — Any extra loose materials or dirt particles on the filter.
- Discoloration* — Any apparent discoloration that might be evidence of contamination.
- A filter with any imperfection not described above, such as, irregular surfaces or poor workmanship.

9.3 Filter Weighing and Equilibration

Filters and balance must be handled in a controlled environment. For conditioning/equilibration of filter prior to weighing, place blank or exposed filters in conditioning chamber with temperature maintained at $25 \pm 2^\circ C$ and RH at 45 ± 5 percent for at least 24 h. As loose dust particles can affect the measurement, the filters should be kept in covered petri-dishes at all times. Store pre-weighed filters in covered dishes in a clean area. Filters must be equilibrated in the same environmental conditions prior to the pre and post-sampling weighing.

Filters should be weighed immediately after removal from the conditioning chamber maintaining temperature of balance room at $25 \pm 2^\circ C$ and RH at 45 ± 5 percent during the weighing process. Use clean, gloved hands and handle the filters carefully with blunt tweezers (*see 6.3*). Tare the balance to zero without any weight on pan. Place filter on the pan of the balance and read the weight. Record the filter ID and weight. Once the difference between consecutive readings is less than 0.015 mg, the filters are considered stabilized.

NOTE — Verify the zero after weighing of every ten filters. After every 10 filters it is recommended that the weight of a standard weight be verified to check for drifts in the balance.

After initial weighing, filters shall be used for sampling within 30 days. Post sampling conditioning and weighing of filters shall be completed within 10 days after the end of sampling period.

9.4 A system of filter blanks shall be maintained. Filter blanks must be transported to the site and processed almost exactly as the actual filters (except the sampler would not be operated). Filter blanks are an important quality control procedure and consistent blank weights will increase the confidence level of actual sample weights. A lot blank check can also be performed.

9.5 Loading Filters into Cassettes

Avoid direct contact with filters at all times. To facilitate the same, filters are placed in plastic cassettes which are in turn placed in the filter holder of the sampler. To load a pre-weighed filter in a cassette, open the cassette by prying open the two halves. Carefully place the filter in the cavity of the lower half and place it by pressing the top part. Immediately place the loaded cassette in a covered carrier / container for transporting to the field site.

9.6 Field Sampling

Use the sampler manufacturer's instructions to setup the instrument. Place a filter cassette (with pre-weighed filter) in the filter holder of the sampler. Switch on the sampler and check the sampling rate. For systems having air volume totalizer, record the initial volume reading else record the time totalizer reading. Allow the sampler to operate for the desired sampling period. Before switching off the sampler, check that the flow rate has been maintained within acceptable limits as per 8.1. Record the volume totalizer or time totalizer reading to determine the total volume of air sampled. Remove the filter cassette from the filter holder assembly of the sampler and immediately place it in a covered container for transportation to the laboratory for post sampling weighing.

9.7 Period of Operation

Samples shall be collected for 24 h period in line with NAAQS (National Ambient Air Quality Standards), India. However, in areas of frequent power failure or difficult site conditions a sample that has been collected for at least 16 h (out of the 24 h period) may also be accepted as a valid sample.

9.7.1 In areas with high PM concentration, it has been observed that particularly in the presence of high humidity/fog the PTFE filter membrane gets rapidly choked and it becomes impossible to determine a 24 h average concentration via one continuous sampling. In such situations a 24 h value may be reported by taking an arithmetic mean of 2 or more samples covering a period of at least 16 h out of the 24 h duration.

10 QUALITY ASSURANCE MEASURES

10.1 Leak Check

Follow the sampler manufacturer's manual to setup the leak check apparatus. External leak check should be performed periodically to ascertain that there is

no leakage of air that affects the performance of the system. It is recommended that a leak check must be performed after any maintenance activity on the sampler and after the instrument has been moved to a new site. The procedure should include a method that checks the system's ability to hold a vacuum of at least 75 cm water column for a period of 10 min with a drop in pressure that corresponds to a leak rate of less than 80 ml/min.

10.2 Calibration

Periodic calibration of the sampler and balance must be performed as per the calibration plan prepared by laboratory at such intervals as determined on the basis of use. The laboratory shall have masters of F2 class or better (weight box for balance and an independent flow rate measuring device traceable to national primary standards with an accuracy of ± 2 percent) to perform intermediate checks at a single point for balance and sampler. If during any intermediate check, deviation of more than ± 4 percent of the designed flow rate is observed, calibration must be performed again.

NOTE — Calibrations shall be carried out by accredited laboratories following system as per IS/ISO 17025 or by laboratories having traceability system. For balance, on intermediate checks, if the standard deviation is greater than ± 3 micrograms, and the R^2 is not better than 0.995, the balance should be carefully inspected and submitted for maintenance and recalibration by an IS/ISO 17025 accredited calibration laboratory. The overall frequency of calibration shall in no case be more than 2 years.

The quality of the entire sampler system to determine mass concentration of $PM_{2.5}$ is based on the dust segregation system and $PM_{2.5}$ impactor as given in 7.4 and 7.5. The model of $PM_{2.5}$ sampler shall thus be approved for 2.5 μm cutoff size from National Measurement Institute (NMI) or any other institute authorized by the National Standards Body of India.

11 CALCULATION

11.1 Calculation of Volume of Air Sampled:

$$V = Q \times t$$

Where,

V = volume of air sampled, in m^3 ;

Q = average flow rate, in m^3/h ; and

t = total sampling time, in h.

11.2 Calculation of $PM_{2.5}$ Concentration in Ambient Air:

$$PM_{2.5} \text{ (as } \mu g/m^3 \text{)} = ((W_2 - W_1) \times 10^6) / V$$

Where,

$PM_{2.5}$ = Mass concentration of fine particulate matter less than 2.5 microns diameter, in $\mu g/m^3$;

W_1 = initial weight of the filter, in g;

W_2 = final weight of filter, in g;

V = Volume of air sampled, in m^3 ; and

10^6 = conversion factor of g to μg .

BIBLIOGRAPHY

1. 40 CFR, Part 50, Appendix L – USEPA Reference Method for the determination of Fine Particulate Matter as $PM_{2.5}$ in the atmosphere.

ANNEX A

(Foreword)

COMMITTEE MEMBERS RESPONSIBLE FOR DEVELOPMENT OF STANDARD

<i>Organization</i>	<i>Representative(s)</i>
In personal capacity	DR (MRS) GAURI G. PANDIT
Avantha Centre for Industrial Research and Development, Yamuna Nagar	DR NISHI KANT BHARDWAJ DR SUNIL KUMAR (<i>Alternate</i>)
Bhabha Atomic Research Centre, Mumbai	DR A. VINOD KUMAR DR S. K. SAHU (<i>Alternate</i>)
Central Institute of Mining and Fuel Research, Dhanbad	DR R. C. TRIPATHI DR N. K. SRIVASTAVA (<i>Alternate</i>)
Central Pollution Control Board, New Delhi	SHRI V. K. SHUKLA DR R. C. SRIVASTAVA (<i>Alternate</i>)
Confederation of Indian Industry, New Delhi	SHRI SHIKHAR JAIN MS NANDINI KUMAR (<i>Alternate</i>)
Delhi Technological University, New Delhi	DR RAKESH MEHROTRA DR ANUBHA MANDAL (<i>Alternate</i>)
Director General, Factory Advice Service & Labour Institute, Mumbai	DR BRIJ MOHAN MS DHANASHREE ACHREKAR (<i>Alternate</i>)
Ecotech Instruments, Greater Noida	DR RAJENDRA PRASAD
Envirotech Instruments Pvt Ltd	DR BALBIR SINGH SHRI HARSH KUMAR DIXIT (<i>Alternate</i>)
Environment Protection and Training Research Institute, Hyderabad	DR N. RAVEENDHAR
Gujarat Pollution Control Board, Gandhinagar	DR D. N. VANSADIA
Green Economy Initiatives Pvt Ltd, Mohali	DR R. S. SAINI
Himachal Pradesh State Pollution Control Board	DR T. B. SINGH DR MANOJ CHAUHAN (<i>Alternate</i>)
In personal capacity	SHRI SANJIB KU. GOSWAMI
In personal capacity	DR S. K. TYAGI
In personal capacity	DR H. K. PARWANA
Indian Association for Air Pollution Control – Delhi Chapter	DR J. S. SHARMA
Indian Chemical Council, Mumbai	DR RAKESH KUMAR DR PRANAV K. TRIPATHI (<i>Alternate I</i>) SHRI J. I. SEVAK (<i>Alternate II</i>)
Indian Institute of Toxicology Research, Lucknow	DR S. C. BARMAN
Jharkhand State Pollution Control Board	SHRI R. N. KASHYAP
Maharashtra State Pollution Control Board	DR A. R. SUPATE DR V. R. THAKUR (<i>Alternate</i>)
National Environmental Engineering Research Institute, Delhi Centre	DR S. K. GOYAL DR P. K. LABHASETWAR (<i>Alternate</i>)
National Council for Cement and Building Materials, Ballabhgarh	DR N. K. TIWARY SHRI ANAND BOHRA (<i>Alternate</i>)
National Physical Laboratory, New Delhi	DR SHANKAR AGARWAL
NTPC Ltd, New Delhi	SHRI V. RAVI BABU SHRI SUDHIR DAHIYA (<i>Alternate</i>)

<i>Organization</i>	<i>Representative(s)</i>
Sriram Institute for Industrial Research, New Delhi	DR MUKESH GARG SHRI RAJNEESH KU SHARMA (<i>Alternate</i>)
Fertilizer Association of India, New Delhi	SHRI SATYA SUBRAHMANYAM SHRI MANISH GOSWAMI (<i>Alternate</i>)
Thermofisher Scientific	MS JAYA NANGIA SHRI ANANT GHADIGAONKAR (<i>Alternate</i>)
UNIPHOS Envirotronics Pvt Ltd	SHRI VIJAY PANDEY DR R. C. NAIK (<i>Alternate</i>)
West Bengal Pollution Control Board, Kolkata	DR UJJAL KU MUKHOPADHYAY SHRI SHYAM KU ADHIKARI (<i>Alternate</i>)
Director General, BIS	SHRI U. K. DAS, SCIENTIST E AND HEAD, CHD [REPRESENTING DIRECTOR GENERAL (<i>Ex-officio</i>)]

Member Secretary

KU KRETI DAS
Scientist B (CHD), BIS

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402

Website: www.bis.gov.in

Regional Offices:

Telephones

Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	{ 2323 7617 2323 3841
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